T10D11 – Oxidation of Ethanol Lab Practical

Aim: The purpose of this experiment is to use the quick-fit apparatus to isolate and observe the various products of the oxidation of ethanol.

Lectured Material: Topic 10.4 (Alcohols) and the mechanism for the oxidation has already been discussed.

Introduction: As you are aware, the oxidation of an alcohol can produce various compounds depending on the original state of the compound (primary, secondary, tertiary) and the conditions the product is subjected to. Several tests have been outlined to help identify the product. You will complete the lab and write up a Conclusion/Evaluation for this experiment.

The tests you will use are as follows:

Fehling's Solution:
You will use Fehling's solution in some of your tests. This solution contains a blue copper (II) compound which, in the presence of a reducing agent, changes to a red copper (I) compound.

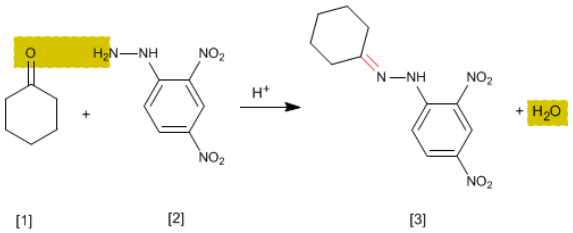
$$Cu^{2+}\text{ (blue)} + e^- \rightarrow Cu^+ + Cu_2O\text{ (red)}$$

Tollens' Reagent:
You will also use an ammonia solution of silver oxide (Tollens' reagent) which, in the presence of a reducing agent, produces a 'silver mirror' (and/or a grey precipitation):

$$Ag^+ + e^- \rightarrow Ag\ (s)$$

Each of these tests confirms the presence of a reducing agent.

Test 2.4-dinitrophenylhydrazine:
This is a specific analytical test aldehydes and ketones. Carbonyls [1] react with 2,4-dinitrophenylhydrazine [2] forming phenylhydrazones [3] yellow precipitate. The appearance of precipitate is an indicator of the presence of carbonyls in the middle.



[1] [2] [3]

Most aldehydes and ketones are produced dinitrophenylhydrazones insoluble solids. At first, the precipitate can be oily and the rest become crystalline.

Baking Soda for Carboxylic Acids: The reaction of a carboxylic acid with baking soda will produce CO_2, all others will remain unchanged.

Hazard Warnings:
Ethanol is flammable, you must:

- Keep the stopper on the bottle when possible
- Keep the bottle away from flames
- Wear goggles

Concentrated H_2SO_4 is very corrosive and reacts vigorously with water, you must:

- Wear safety goggles
- Cover spills with sodium bicarbonate (baking soda) and mop up minor spillages with plenty of water
- Dispose of unwanted residues by cooling and pouring slowly into an excess of water

Tollens' reagent for the Silver Mirror test becomes explosive if dry, you must:

- Wash away the solution from experiments A and B immediately once you have finished

Potassium dichromate is a powerful oxidizing agent and can damage the skin. You must,

- Wear goggles and gloves if possible

Procedure:

A: Mild Oxidation (Oxidation and Distillation):

1. Into a pear-shaped flask, pour 10cm^3 of 1.0M H_2SO_4. Using a small wide-stemmed funnel, add 3.0g of $K_2Cr_2O_7$ and 2-3 boiling chips ($CaCO_3$)
2. Swirl the flask gently until all the potassium dichromate has dissolved.
3. Slowly add 5 cm^3 of ethanol and swirl to mix.
4. Set up the apparatus shown. Ensure that the water enters the condenser from the bottom and leaves at the top.
5. Heat very gently until 2-3 cm^3 of liquid has distilled over.
6. Keep the distillate and test it in the following ways:
    a. Waft (smell) cautiously (compare with ethanol)
    b. **Fehling's Test:** Transfer about 1cm^3 of the distillate to a test tube. Add about 1 cm^3 of Fehling's solution 1 followed by 1cm^3 Fehling's solution 2. Boil gently. Note your observations.
    c. **Silver Mirror (Toliens') Test:** Pour about 5cm^3 of 0.05M $AgNO_3$ solution into a boiling-tube. Add one drop of 2.0M NaOH solution. Drop by drop, add $NH_3(aq)(NH_4OH)$ until the precipitate disappears. Add 2-3 drops of the distillate and warm the tube in a beaker containing hot water. Note your observations.
        i. **DO NOT** keep this solution, it becomes explosive upon evaporation!!!
    d. **2.4-dinitrophenylhydrazine Test:** Dissolve 2 drops of the compound to be investigated in 2 ml of ethanol 95% and add the solution to 3 ml of 2.4-dinitrophenylhydrazine reagent (previously prepared by the HS lab Technician). Shake vigorously and if a precipitate is formed immediately, let the solution stand for 15min.[i]
        i. PS: you should make the test control, put ethanol into a tube and another tube ketone to see how the test is positive and how is negative.

B: Further Oxidation (Under reflux):

1. Into a pear-shaped flask, pour 10cm^3 of 1.0M H_2SO_4. Through a wide-stemmed funnel add 5.0g $K_2Cr_2O_7$ and 2-3 boiling chips ($CaCO_3$).
2. Swirl the flask gently until all the potassium dichromate has dissolved.
3. With care, add 2cm^3 concentrated H_2SO_4.
4. Cool the flask under a running tap.
5. Set up the apparatus shown, preferably in a fume-hood.
6. Drop by drop, add 1cm^3 of ethanol down the condenser.
7. Boil gently under reflux for 20 minutes.
8. Rearrange your equipment so it is set up as a distillation apparatus as in Part A.
9. Distill 2-3 cm^3 of liquid.
10. Test the distillate as follows, and note your observations.
    a. Smell cautiously (compare with ethanol)
    b. Add a drop to moistened universal indicator paper
    c. Add a few drops to about 1.0g $Na_2CO_3(s)$. [ii]
    d. Complete the Baking Soda test, drop a small amount inside a test tube containing each material.

Assessment:

You will be graded on Conclusion/Evaluation on the IB scale:

1. Concluding	2. Evaluating Procedure(s)	3. Improving the Investigation
C P N n/a • Results are repeated at beginning • Major Results • % Error • Uncertainty • Units • Refer back to graph (repetitive, yes) C P N n/a • Restate and respond to the research question C P N n/a • Literature values are referenced (cited using MLA) C P N n/a • Describe the **chemistry** behind the experiment • Relevant IB Curriculum Material • **JUSTIFICATION** of results or lack of C P N n/a • Conclusion is based on your results (specifically) C P N n/a • Uncertainty compared to percent error	C P N n/a • Explain difference between expected/observed C P N n/a • Comment on the reliability of procedure C P N n/a • Can the results be trusted (outliers) – what would happen if the outliers were thrown out? C P N n/a • **Table 9a** – list errors/problems/limitations • Random or Systematic Error • Discuss Accuracy vs Precision C P N n/a • **Table 9b** – state impact of each error on results	C P N n/a • **Table 9c** – Improvement to minimize errors C P N n/a • New Equipment (name, precision, location) C P N n/a • Manipulation of procedures (and why) C P N n/a • Suggestions to improve systematic errors if possible C P N n/a • Suggestions (specific) to improve random errors NOT FOR IB CRITERIA – but required for me ☺ C P N n/a • Provide at least one relevant extension for the lab. This can be simply studying a different variable, testing the effect of an error source, real world examples, vocational (job) applications, possible extended essay topics, etc.
C = 2 P = 1 N = 0	***C = 2 P = 1 N = 0***	***C = 2 P = 1 N = 0***

[i] -Systematic identification of organic compounds, authors: Shriner-Fuson-Cutino . Limusa Noriega Publishers, page 142

- http://www.quimicaorganica.org/aldehidos-y-cetonas/ensayo-de-la-2-4-dinitrofenilhidrazina . html

[ii] Chris Goodman. ECA 2009.